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Procedia Social and Behavioral Sciences

Procedia - Social and Behavioral Sciences 195 (2015) 2457 – 2465

World Conference on Technology, Innovation and Entrepreneurship

Suitability of Biochar Produced from Biomass Waste as Soil Amendment

Nur Shahidah binti Ab Aziz[a]*, Mohd Adib bin Mohd Nor[b], Shareena Fairuz binti Abdul Manaf[b], Fazlena Hamzah[b]

[a]*Faculty of Chemical Engineering, Pasir Gudang Campus, Jalan Purnama, 81750 Masai, Malaysia*
[b]*Faculty of Chemical Engineering, UiTM Shah Alam, 40450, Shah Alam, Malaysia*

Abstract

Conversion of biomass waste to a useful product has been extensively studied to compensate its abundance in quantity and combat current environmental pollution issue. Of the biomass listed comes from agriculture industry; empty fruit bunch (EFB). This study was conducted to investigate the suitability of this waste as soil amendment in improving soil quality. The waste is converted to biochar by mean use of pyrolysis technique. Pyrolysis temperature was varied to 400°C, 500°C and 600°C and the physiochemical properties were analyzed. The results obtained from this study indicate that as the pyrolysis temperature increases, the BET surface area of pores available on EFB biochar decreases. Besides, only small amount of biochar can be produced at high pyrolysis temperature. Several bands or peak diminished from the FTIR spectra of EFB biochar as the temperature rises. From the result also, it was observed biochar that was produced at 400°C pose a characteristic of high yield (50.60%), high fixed carbon content (31.89%) and with suitable morphological features such as high pore volume with better surface area which is important for carbon sequestration. At higher pyrolysis temperature, the crystalline structure of the biochar gradually decreases due to the lower in peak value of cellulose and lignin wavelength in XRD analysis. Besides, it is very alkaline (pH 10.88) which is suitable in neutralizing soil acidity and it contains high CEC value (35 meq/100g) which is believed suitable to increase soil fertility.

Peer-review under responsibility of Istanbul Univeristy.

Keywords: Biochar; Empty Fruit Bunch, Pyrolysis, Temperature

* Corresponding author. Tel.: +607-3818530.
E-mail address: shahidah_aziz@johor.uitm.edu.my

Peer-review under responsibility of Istanbul Univeristy.
doi:10.1016/j.sbspro.2015.06.288

1. Introduction

Malaysia has been one of the world's largest producers and exporters of crude palm oil. Almost 70% of the volume from the processing of fresh fruit bunch is removed as wastes in the form of empty fruit bunch, palm kernel shells and palm oil mill effluent (Zafar S., 2013). Today, new research has been conducted on the palm oil tree and new technologies has been developed which utilizes the usefulness of a single palm oil tree for the benefits of human and environment. Other than being utilized in biofuel production, EFB also can be pyrolyzed to be converted into biochar using pyrolyzer.

Biochar can be produced either by slow pyrolysis or fast pyrolysis, similar as the method to produce charcoal. However, the vast amount of EFB accumulation due to current demand of biofuel seems to be a problem among farmers and palm oil plantation developers. Heavy accumulation of EFB need to be managed properly as this may cause problems such as damage to field roads and frequent upgrading of harvesting paths which can be costly due to poor management of EFB in palm oil field (Shafie et al., 2012). Furthermore, the usage of pure charcoal will cause great impact on the environment such as greenhouse gases emission, global warming crisis, thinning of ozone layer and eventually, altering the world climate.

Recent studies has discovered that biochar might have the potential to reduce the greenhouse gases emission produced from combustion of charcoal. Based on several research conducted in United States of America (USA), Colombia, Kenya and Australia, it is generally accepted that biochar has a highly stable form of carbon and has the potential to perform the sequesteration of carbon dioxide at billion-ton scale for 30 years time (Sohi S. et al., 2009). Another unique ability of biochar is that it can increase cation exchange capacity (CEC) and pH of soil (Shafie et al., 2012), thus increasing soil moisture, total nitrogen and phosphorus ions, promotes root development (Chan et al., 2008), minimize soil erosion and nutrient leaching during drought (Lorenz, 2007).

The yield and quality of biochar is critically influenced by the type of biomass used, chemical and structural composition of biomass, pyrolysis temperature, process heating rate and type of pyrolysis reactor being used (Shafie et al., 2012). Among these factors, the pyrolysis temperature opposes significant changes on the physical and chemical properties of EFB biochar. Thus, this study is carried out to investigate the effect of temperature on the ability and performance of EFB biochar as biocompost. More importantly, this biomass has yet to be fully utilized economically and commercialized in Malaysia despite of its benefits to human and environment as well.

2. Methodology

2.1. Materials preparation

Fresh shredded empty fruit bunch was collected at Meru, Klang and placed in air tight plastic bags to keep the moisture content of the EFB. The EFB was stored in the laboratory at temperature around 15°C to maintain the freshness of EFB. Fresh EFB was dried in the oven at temperature of 105°C for 3 hours in order to avoid soot formation during pyrolysis process (N. Claoston et al., 2014). The colour of shredded EFB has changed from dark brown to light brownish in colour due to the heat applied during drying process. The dried EFB was then stored in sample plastic bag prior to pyrolysis process.

2.2. Equipment and method for analysis

Yield of biochar produced was calculated by dividing the mass of EFB biochar produced with the mass of EFB after dried before pyrolysis process. Moisture content was analysed using Moisture Analyzer and ash content was analysed after burning process in furnace at 815°C for more than 3 hours.

The physicochemical properties of biochar produced was analysed in term surface area, particle morphology, thermal stability, presence of elements, pH value and total cation available. Brunauer-Emmet-Teller Surface Area (SBET) is performed to calculate the surface area of EFB biochar pores in the whole structural component by using the adsorption data in relative pressure ranges from 0.05-0.20. This technique is carried out by applying highly purified nitrogen gas on the sample. The biochar sample will be heated at 150℃ for 2 hours under vacuum condition. After the heating process, the nitrogen gas is allowed to be aerated through the sample. From here, the surface area of biochar sample is determined by measuring the adsorption isotherm of the sample towards the nitrogen gas (Sukiran M. A. et al., 2009).

Scanning electron microscopy (SEM) is an instrument which allow user to view the macroporosity and biochar particles morphology after pyrolysis process. The biochar sample was attached to an aluminum stub as a general setup. The stub was then placed in a sputter coater in order to cover the biochar sample with gold. The coating was performed in vacuum condition with pressure of 0.1 mbar for 3 minutes and current of 35 mA. During this process, the images of biochar sample surface were captured and transferred into the control system and displayed on the monitor. After coating process, the stub was placed in the sample holder of SEM and the images of sample can be viewed from the connecting monitor (Shafie S. T. et.al., 2012).

The thermal stability and decomposition of EFB biochar were measured by using thermogravimetric analyzer (TGA). The samples of EFB biochar which pyrolyzed at the ranges of 400°C, 500°C and 600°C were analyzed. About 5 mg of biochar sample from different temperatures which are at 400℃, 500℃ and 600℃ were placed in aluminium pan of the TGA instrument. The samples are heated until the temperature reaches 600℃ with a heating rate of 10°C/min in the presence of air. The losses in weight of biochar sample were recorded to construct TGA and DTG curves. The presence of specific functional groups in EFB biochar was identified using Fourier Transform Infrared Spectrophotometer (FT-IR) (Perkin Elmer, Spectrum GX, UK) (N. Abdullah et al., 2011) between 400 cm^{-1} to 4000 cm^{-1} of infrared wave length by thermally compressing the samples into a thin film.

The pH is an important property of the soil, which influences the types of plants and microbes to thrive, and the availability of nutrients to be absorbed (Lee Y. et al., 2013). However, the pH of biochar is determined in this case instead of soil pH. The pH of biochar is determined by dissolving 1 g of biochar sample with deionized water with a ratio of 1: 100 (biochar: deionized water). The mixture was stirred and heated at 90°C for 20 minutes prior to being sieved using filter paper. The pH of the solution was measured using pH meter.

Cation exchange capacity (CEC) is a measure of the maximum quantity of total cation available in soil being tested. CEC is invented in order to measure the soil fertility, nutrient retention capacity and the tendency to protect groundwater from cation contamination. The value of CEC of biochar was estimated by using ammonium ion (NH_4^+) exchange method. Firstly, about 0.5 g of biochar was rinsed twice with 25 mL of 1M ammonium acetate (CH_3COONH_4) at pH 7 in order to saturate the exchange sites. Any excess solution from the biochar sample was washed with 10 mL of 95% ethanol. The adsorbed ammonium (NH_4^+) ion was extracted by rinsing the biochar with 25 mL of 0.1M potassium chloride (KCl) twice. The presence of NH_4^+ in the solution is tested by titration with 0.05M NaOH in the presence of 5 mL formalin and phenolphthalein (Uddin M. A. et al.). The amount of NH_4^+ ions contained in the solution can be determined by applying the CEC calculation by AOAC (1975) and Maclean A. J. et al. (1964).

XRD which stands for X-ray diffraction analysis is performed by directing a single beam of X-ray onto a crystal at a specific angle and the beam will be reflected on the sample being tested. The X-ray diffractometer was equipped with Ni-filtered Cu-Ka radiation (k = 1.5406 Å) at an accelerating voltage of 40 kV and an emission current of 40 mA to direct X-ray beam on the sample. The X-ray diffraction pattern range was set from 10° to 70° and this analysis was conducted for about 30 minutes at a speed of 2.5°/min.

3. Analyses and Results

3.1 Yield of bochar

Table 1.1. Yield of EFB biochar at different operating temperature

Temperature (°C)	Percentage of biochar yield (%)
400	50.60
500	45.81
600	38.67

From the results obtained, the yield of biochar seems to decrease as the temperature increases. Any increase in temperature will cause the volatile components such as water and nutrients exist inside EFB to slowly evaporate into the atmosphere thus, leaving ash and char as the sole product of the pyrolysis process. This shows that the yield of biochar depends on the temperature used during pyrolysis process. Nevertheless, as the temperature increase, the heating value and surface properties of biochar are improved (M. Amutio et al., 2012).

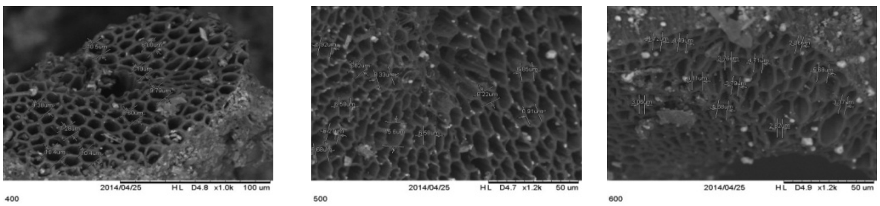
3.1. Surface area and pore sizes

From Table 1.2, the values of BET surface area for three samples of EFB biochar are 4.139 m^2/g, 7.658 m^2/g and 4.172 x 10^{-1} m^2/g respectively. These value of BET surface area shows that biochar at 500°C has a larger surface area compared to that of 600°C. The result shows that the surface area and structure of biochar becomes more ordered arrangement and wider pore volume as the pyrolysis temperature increases. However, during 600°C, the biochar structure starts to experience cracking, shrinkage and rupture due to excess heating process. This explains the decrease in value of biochar surface area at 600°C.

Table 1.2. Porous properties of EFB biochar produced at different operating temperature

Sample	BET surface area (m^2/g)	Pore volume (cc/g)	Average pore diameter (Å)
EFB biochar 400°C	4.139	-5.029 x 10^{-2}	-4.860 x 10^{2}
EFB biochar 500°C	7.658	5.908 x 10^{-6}	3.086 x 10^{-2}
EFB biochar 600°C	4.172 x 10^{-1}	3.30 x 10^{-4}	31.93

The total pore volume and average pore diameter for EFB biochar at 400°C, 500°C and 600°C obtained from BET analysis are -5.029 x 10^{-2} cc/g and -4.860 x 10^{2} Å, 5.908 x 10^{-6} cc/g and 3.086 x 10^{-2} Å, 3.30 x 10^{-4} cc/g and 31.93 Å. Generally, as the pyrolysis temperature increases, the surface area of biochar samples increases as well due to the removal of volatile material which results in increased macropore volume (Ahmad M. et al., 2012) (Claoston N. et al., 2014). This can be proven based on the results of pore volume and average pore diameter obtain in which the EFB biochar produced at 600°C possess larger pore volume and wider pore diameter compared to biochar produced at 500°C followed by biochar produced at 400°C.



(a) EFB 400°C (b) EFB 500°C (c) EFB 600°C

Fig. 1.1 Morphology of EFB biochar particles at 400°C, 500°C and 600°C

Several pores with uniform sizes are measured in order to obtain the average pore size of EFB biochar at that temperature. This value of average pore size is crucial in investigating the effect of pyrolysis temperature on the uniformity of biochar structure. The average micropore size of EFB biochar at 400°C, 500°C and 600°C are 8.84 μm, 7.71 μm and 4.25 μm. Based on this observation, it seems that the micropore size of EFB biochar particles decreases while the macropore size increases as the pyrolysis temperature increases.

At temperature of 400°C, the tissue and cellulosic components of EFB has not been fully degraded. Therefore, the pore of biochar particles at 400°C is seen to be more stable and solid compared to that of biochar particles at 500°C and 600°C. As the temperature increases, more volatile matter will gradually vaporizes, causing the pore of biochar particles to be hollow. At 600°C, the initiation of degradation and cracking starts to occur on the structure of biochar particles due to lack of fluid components left. This shows that the structure of biochar degrades or ruptures due to the increase in temperature. This results in the formation of irregular structure of biochar, thus reducing the uniformity of biochar pore structure. The biochar formed at high temperature is more brittle compared to biochar formed at lower temperature as it cannot withstand pressure due to its fragile structure (Claoston N. et al., 2014).

3.2. Thermal stability

The EFB biochar sample produced at 400°C only starts to experience weight loss during the temperature of 265°C. This indicates that the sample does not contain any more moisture. Low moisture content will increase the availability of fixed carbon contained in the sample thus, increasing soil fertility. At 265°C to500°C, the mass of biochar decrease by 65.81% due to the vaporization of volatile matter and degradation of cellulose and lignin components as reported by Tröger, Richter & Stahl (2013). Since the ash content can only be obtained at temperature above 900°C, the residue obtained at the final analysis indicates the fixed carbon content of the sample which is about 31.89% from the original weight of sample.

Similar to EFB biochar sample produced at 400°C, the results obtained for sample produced at 500°C show absence of moisture content available inside sample. However, the sample loses about 74.22% of its weight at temperature between 225°C to 500°C, showing the sample initiates degradation process at temperature lower than the degradation process that occur in sample produced at 400°C. From here, it can be assumed that the sample produced at 500°C has a lower thermal stability compared to that of 400°C, indicating that the sample can only retain its structure until the temperature reaches 225°C before it starts to turn into ash. The carbon content obtained is 18.92% from the initial weight of sample which is lower than that obtained in sample produced at 400°C.

The EFB biochar sample produced at 600°C loses about 6.62% of its weight from temperature of 25°C to 100°C. This shows that about 6.62% of the sample weight consists of moisture and water vapour. The presence of moisture inside the sample may be due to prolong storage of sample before conducting analysis thus, allowing the sample to gain any traces of moisture available in the atmosphere to be entrapped in between the pore of the biochar sample. Approximately at 305°C to 500°C, the sample experiences a significant amount of weight loss which is 63.62% from its initial weight, leaving only 27.73% of carbon compounds.

3.3. Proximate Analysis

Table 1.3. Properties of raw EFB and EFB biochar at different pyrolysis temperatures

Properties	Raw EFB	EFB biochar		
		400°C	500°C	600°C
Yield (wt. %)	-	50.60	45.81	38.67
Moisture content (%)	33.29×10^{-3}	0.00	0.00	6.62
Volatile matter (%)	55.83	65.81	74.23	63.62
Ash content (%)	14.36	-	-	-
Fixed carbon content (%)	30.25	31.89	18.92	27.73

The results of proximate analysis of raw EFB sample is compared with the TGA results of EFB biochar at 400C, 500C and 600C. Table 1.3 summarized the properties and chemical characteristics of raw EFB and EFB biochar produced at different operating temperatures. Any biochar with low amount of moisture content, low volatile matter content, low amount of ash produced and high fixed carbon content is suitable to be used as soil amendment. Since the ash content can only be obtained at temperature above 900°C, it is neglected from discussion.

Based on the results obtained, EFB biochar produced at 600°C contain slight amount of moisture which is 6.62% compared to that of 400°C and 500°C. High amount of moisture will fill up the void space in between the pore surface of the biochar thus, causing the biochar to experience immediate rupture due to the structure's fragility. However, EFB biochar produced at 600°C liberate the least amount of volatile compounds among the three samples which is 63.62%. High amount of volatile matter in biochar is a disadvantage since it will reduce the amount of carbon available which is the main component for carbon sequestration. Besides, lots of heat energy is required to remove the volatile matter in EFB sample to form biochar thus, increasing the cost for biochar production. By comparing the fixed carbon content properties, EFB biochar at 600°C possess moderate amount of carbon content among the three samples thus, making it the most stable sample with high carbon content.

3.4. Presence of functional group

Based on the results obtained, the EFB biochar produced at 400°C consists of 17 functional groups such as ammonium ions, carboxylic acid, nitramines, carboxylate ions and others. About 13 different functional groups are present in EFB biochar produced at 500°C while 20 funtional groups are spotted on EFB biochar sample produced at 600°C. Among the three samples, there a few types of functional groups that exist consistently regardless of the temperature being applied on the EFB sample such as carboxylate ions, boron compounds (B-O and B-N), sulphur compound, nitrate ions, C-H bending molecules (OCOCH), O-H bending and halogen compound.

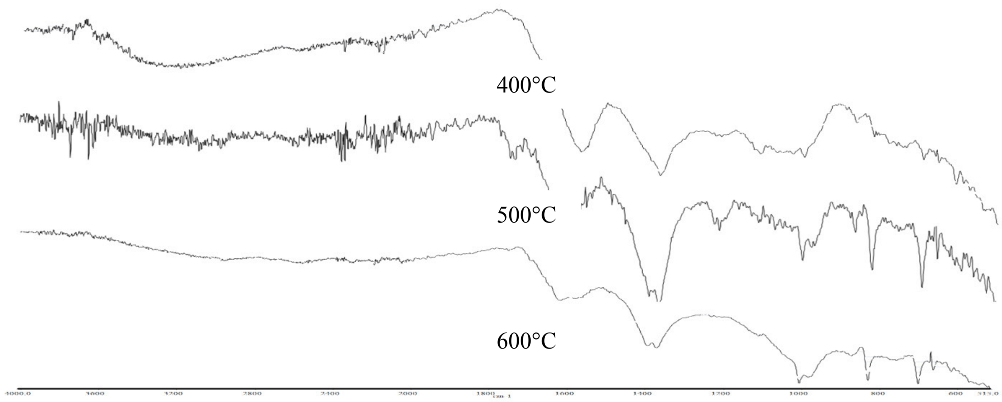


Fig. 1.2. FTIR spectra of EFB biochar produced at different operating temperatures

From Figure 1.2., the intensities of the bands or wavelengths of the functional group seem to decrease with increase in temperature being applied on the EFB samples. This indicates the loss of water contained inside the EFB samples due to increasing temperature (Yaakob Z. et al., 2012). At wavelength 3600-3100 cm^{-1}, the absence of crystallized water in EFB biochar sample produced at 500°C and 600°C proves the pattern of FTIR spectra in Figure 4.7. Other functional groups such as ammonium ions and carboxylic acid bonds can be observed in EFB biochar produced at 400°C at the same peak with crystallized water which is at 3198.21 cm^{-1}. Various elements such as N-H bending, carboxylate ions, amine salts, nitramines and saturated nitroso compounds are available at the spectra between 3200-1370 cm^{-1} at the same temperature.

Based on the data obtained from Figure 1.5, it can be seen that pyrolysis process at high temperature causes the EFB to experience shrinkage, degradation and rapid dehydration due to the presence of heating element. The disappearance of O-H bending as temperature increases indicates the initiation of cellulose, hemicelluloses and lignin degradation and depolymerization at high temperature (Cantrell et al., 2012). Besides, the sudden appearance of silicon in EFB biochar at 600°C shows the initiation of biochar structural modification due to the increasing in temperature which causes the degradation of cellulose and lignin, exposing the abundant amount of silicon compounds entrapped inside the biochar (Claoston N. et al., 2014).

3.5. pH Analysis

Table 1.4. pH value of EFB biochar at different operating temperature

Temperature (°C)	pH value
400	10.88
500	10.73
600	10.77

Table 1.4 shows the pH value of EFB biochar obtained at three different pyrolysis temperatures which are 400°C, 500°C and 600°C. The EFB biochar produced at 400°C possess the highest pH value which is 10.88 compared to the other two temperatures. From the results obtained, the pH value of EFB biochar remains constant as the temperature increases. Since the EFB biochar produced from three different temperatures have high reading of pH values, it can be assumed that the EFB biochar is in highly alkaline condition. High pH value of EFB biochar makes it suitable to reduce the acidity of soil (Udoetok I. A., 2012). Besides, high pH value of EFB biochar will result in better breakdown of soil pollutants and increase the availability of soil nutrients (Udoetok I. A., 2012). These advantages of highly alkaline EFB biochar are supported by Udoetok I. A. in his study. This shows that EFB biochar is suitable to be used for soil amendment in order to reduce the acidity of soil and increase the availability of soil nutrients.

3.6. Cation Exchange Capacity (CEC)

Table 1.5. CEC value of EFB biochar at different operating temperature

Temperature (°C)	CEC value (meq/100g)
400	35
500	33
600	24

Table 1.5 shows the CEC value of EFB biochar obtained at three different pyrolysis temperatures which are 400°C, 500°C and 600°C. The EFB biochar produced at 400°C possess the highest pH value which is 35 meq/100g compared to the other two temperatures. From the results obtained, the pH value of EFB biochar gradually decreases as the temperature increases. This is because of the oxidation of the aromatic carbon ring and formation of carboxyl groups in biochar as the pyrolysis temperature increase (Liang B. et al., 2006). This statement by Liang B. et al. is supported by McBeath and Smermik whom reported that the aromatization of carbon in the ash of biochars produced at high pyrolysis temperatures might reduce the content of CEC in the biochar.

High CEC value of EFB biochar will enhance the soil fertility (Uddin M. A. et al.). Besides, high CEC value of EFB biochar will result in better soil quality as the presence of CEC will increase the retention ability of nutrients in soil which is good for growing plants (Uddin M. A. et al.). These advantages of highly alkaline EFB biochar are supported by Uddin M. A. et al. in his study. This shows that EFB biochar is suitable to be used for soil amendment in order to reduce the loss of nutrients in the soil thus, increasing the soil fertility as well.

3.7. XRD Analysis

The peaks obtained for each sample are tabulated in Table 1.6. Based on the results obtained, the peak values and peak height of each temperature are in ascending manner as the temperature increases. However, the peak spacing of each peak value gradually decrease as the temperature rises. The elements that can be observed at the peak value of 28° and 40° are silicate minerals such as quartz (SiO_2), hematite (Fe_2O_3) and goethite ($Fe_2O_3.H_2O$) (Yunus N. A. et al., 2013). As mentioned previously, the number of peak values increase due to the rising in temperature of sample. This indicates that more elements contained inside the EFB biochar sample are being detected as the temperature rises. High temperature being applied on the sample causes the sample to experience cellulose and lignin degradation thus, allowing other elements and minerals embodied inside the sample to be exposed and detected by the equipment.

Table 1.6. Peak data of EFB biochar at different operating temperatures

Sample	Peak value (2θ°)	Peak spacing (Å)	Peak height
EFB biochar at 400°C	28.182	3.1639	139
	40.362	2.2328	79
EFB biochar at 500°C	28.261	3.1552	249
	40.497	2.2256	115
EFB biochar at 600°C	28.242	3.1572	251
	40.421	2.2297	124

In a study carried by Parshetti G. K. et al., the peak of 20° to 30° indicates the presence of microcrystalline structure of cellulose in EFB biochar sample (Parshetti G. K. et al., 2013). From Figure 4.10, the XRD pattern seems to expand and more stable as the temperature increases. This result confirms the degradation of cellulose or structural composition of EFB biochar sample at peak of 20° to 30° as being reported by Keiluweit in which sample with higher temperature will progressively lose intensity and become broader, indicating a gradual decrease in cellulose (Keiluweit M. et al., 2010).

4. Conclusion

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From this study, the results pointed that pyrolysis temperature greatly influence the physicochemical properties of EFB biochar. Based on the results of TGA and Proximate analysis, EFB biochar produced at high temperature has lower amount of volatile substance and higher carbon content as the volatile material have been removed during biochar production process. As the pyrolysis temperature increases, the micropore size of EFB biochar decrease while macropore size increases thus, reducing the BET surface area of pores available on EFB biochar. Besides, only small amount of biochar can be produced at high pyrolysis temperature. FTIR spectra of EFB biochar obtained at different operating temperatures revealed variety of inorganic compounds and some similarities in their functional groups. Several bands or peak diminished from the spectra as the temperature rises. Nevertheless, pyrolysis temperature also affects the suitability of EFB biochar to be used as soil amendment. At higher pyrolysis temperature, it can be observed that the crystalline structure of the biochar gradually decreases due to the lower in peak value of cellulose and lignin wavelength in XRD analysis. Furthermore, the pH value of EFB biochar is constant at a value of 10.9 which is in alkaline condition even though the pyrolysis temperature increases. Furthermore, the biochar produced at 400°C possess high CEC value which makes it suitable to increase soil fertility as well as the soil quality.

Acknowledgements

This study was financially supported by Ministry of Education (MOE) of Malaysia through Research Acculturation Grant Scheme (RAGS) (RAGS/2013/UiTM/TK05/6) and Universiti Teknologi MARA, Malaysia.

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